Analytical Methods for the Analysis of and Initially Isolated Formulations of the PMN Substances Required Under Consent Orders:

P-07-0087;

P-08-0200;

P-08-0643, P-08-0642, P-08-0644, and P-08-0664;

P-08-0748 and P-08-0751;

P-09-0245 and P-09-0246;

P-09-0293 and P-09-0294;

P-10-0058, P-10-0059, and P-10-0060;

P-10-0148;

P-11-0091, P-11-0092, and P-11-0093;

P-12-0450

Prepared by:

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Objective

The subject consent orders require the Company to analyze representative samples of the initially isolated formulations of the PMN substances at each manufacturing facility for the analytes specified in Table 3 of the consent orders and report the results at initial commencement of manufacture or import and again if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered. The Company is also required to annually analyze the initially isolated formulations and report these results to EPA, in a cycle complementary to the

In addition to the annual reporting for the initially isolated formulations of the PMN substance, the Company must annually report for the starting material: (1) the average values and range of values, including outlying data, from the routine analyses for the analytes specified in Table 1 of the consent orders and (2) the results of the annual analysis for the analyte specified in Table 2 of the consent orders. This report describes the analytical methods that are used for the required annual analytical reporting.

Materials and Methods

A. Samples Analyzed

1.

Starting in January of 2016 lots of starting material are manufactured and thermally treated at and then used at or shipped to other facilities for conversion into the PMN substance.

2. Initially Isolated Formulations of the PMN Substance

Representative samples of the Initially Isolated Formulations of the PMN substance manufactured or imported by the Company at each manufacturing facility are analyzed at initial commencement of manufacture, and at least annually thereafter. In addition, if any new manufacturing facility is added or if the process of manufacture of the PMN substance or any intermediate thereof is significantly altered, the initially isolated formulation of the PMN substance is analyzed again and reported to EPA. The initially isolated formulations of the PMN substance are analyzed for the analytes specified in Table 3 of the consent orders and reported to EPA.

B. Analytical Methods

1. Analytical Method for

Starting in January of 2016 determination of residual

of the is determined by GC-FID at A GC-FID method is sufficient for quantitative analysis of impurities in to demonstrate compliance with the limits specified in Table 1 of the consent orders; however, a GC-MS method is used on some lots of at for the determination of residual

because some values fall below the limit of detection of the GC-FID method. Either method is adequate for determining compliance with the limits specified in Table 1 of the

consent orders. The limit of quantitation for all analytes specified in Table 1 of the consent orders by GC-FID at is 100 ppm, and the limit of quantitation for all analytes by GC-MS is 2 ppm. The GC-MS method is discussed in detail in Appendix 2.

Starting in January of 2016 for determining compliance with the limits specified in Table 2 of the consent orders, the determination at s achieved by GC-MS analysis of the The limit of quantitation of this method is 2 ppm.

At is quantified in some lots of the by using a modified method based on the published method by Samples are prepared and analyzed with a fortified sample for spike recovery determination. The limit of quantitation for is 2 ppm or 0.5 ppm as is dependent on the sample prep. This method is discussed in detail in Appendix 2.

Results and Discussion

A. Analysis of

A summary of the results, including the range and averages, for analytes specified in Table 1 and Table 2 of the consent orders are presented in specific reports for the starting material.

B. Analysis of the Initially Isolated Formulations of the PMN Substances

The results for analytes specified in Table 3 of the consent orders are presented in specific reports for each consent order.

Conclusion

The analytical methods for the analyses specified in the consent orders for the starting material and all initially isolated formulations of the PMN substances have been presented in this report and will be referenced in all future analytical reports submitted to EPA.

Appendix 1: